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(54) PROCESS FOR THE MANUFACTURE OF PARTLY  
 CRYSTALLINE POLYBUTENE-1

(71) We, CHEMISCHE WERKE HÜLS AKTIENGESELLSCHAFT, a German Company, of 4370 Marl, Germany, do hereby declare the invention, for which we pray that a Patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following Statement:—

The invention relates to the manufacture of partly crystalline polybutene-1. The product manufactured by the process according to the invention is particularly suitable for the manufacture of films.

As is known, films composed of highly isotactic polybutene-1 have the disadvantage that they have a low transparency, very different strength values in the longitudinal and transverse directions and therefore a poor resistance to tear propagation, as a result of which they are not suitable for many fields of use.

The transparency of polybutene-1 film can only be improved slightly by increasing the atactic proportion. If, for example, the atactic constituent is not separated off with the solvent but is left in the product when working up by precipitation with alcohol or by evaporation of the solvent, a polybutene-1 is obtained from which films with only a slight improvement of the transparency are obtained, even when the ether-soluble constituents are 10 to 20%. The resistance to tear propagation is not improved, compared with highly isotactic polybutene-1, and the strengths in the longitudinal and transverse directions are very different, for example longitudinally 24.8 kp/mm and transversely 0.89 kp/mm.

Our copending U.K. Patent Application No. 45513/73 (Serial No. 1437038) has proposed a process for the manufacture of a butene-1 polymer suitable for the manufacture of films by controlled polymerisation, wherein butene-1, alone or with the addition of 0.1 to 15 per cent by weight, relative to butene-1, of a comonomer is polymerised in solution in high strength butene-1 or in a C<sub>4</sub> fraction consisting of butene-1, butene-2 and butane at a temperature of 60 to 120°C by means of a mixed (contact) catalyst of (a)

TiCl<sub>3</sub>.nAlCl<sub>3</sub> (n=0.2 to 0.6), which has been obtained by reduction of titanium tetrachloride with an organo-aluminium compound at a temperature of -10 to +30°C and optionally subsequent heat-treatment at a temperature of 70 to 150°C, and (b) diethyl-aluminium monochloride.

In our earlier U.K. Patent Application No. 5795/74 (Serial No. 1,449,316) we describe and claim a process for the manufacture of a largely amorphous butene-1 polymer by the low pressure method wherein butene-1 is homopolymerised or is copolymerised with from 0.1 to 30 per cent by weight based on butene-1 of one or more other  $\alpha$ -monoolefins as comonomers at a temperature of from 40 to 120°C in bulk (i.e. in high strength butene-1) or in solution in a C<sub>4</sub>-fraction comprising butene-2 and/or butane in addition to butene-1 using a mixed catalyst of TiCl<sub>3</sub>.nAlCl<sub>3</sub> (n=0.2 to 0.6) and an aluminium trialkyl with C<sub>2</sub> to C<sub>4</sub> alkyl groups, at an atomic ratio of Al in the aluminium trialkyl:Ti in the TiCl<sub>3</sub>.nAlCl<sub>3</sub> of 1.2:1 to 5:1 and a concentration of 0.01 to 1 mol of TiCl<sub>3</sub> per litre of total liquid phase.

We have now found that a partly crystalline butene-1 polymer which is particularly suitable for the manufacture of films may be obtained in a simple and economic manner by a process wherein butene-1, alone or with the addition of 0.1 to 10 per cent by weight, relative to butene-1, of a comonomer, is polymerised in a liquid phase which is essentially all butene-1 (i.e. high strength butene-1) or is a C<sub>4</sub> fraction composed of butene-1, butene-2 and butane at a temperature of 60 to 120°C with a mixed catalyst consisting of (a) TiCl<sub>3</sub>.nAlCl<sub>3</sub> (n=0.2 to 0.6) and (b) a mixture of 85 to 99.9 mol per cent, preferably 90 to 99 mol per cent, of a dialkyl-aluminium monochloride and 0.1 to 15 mol per cent, preferably 1 to 10 mol per cent, of an aluminium trialkyl.

By the term "high strength butene-1" in our earlier applications and in the present case is meant in particular a 95 to 99 weight per cent butene-1. The percentage of butene-1 in the C<sub>4</sub> fraction used as an alternative liquid

medium is desirably about 50% by weight or more.

Within the scope of this invention a partly crystalline butene-1 polymer is understood as a product which has a density of 0.90 to 0.91 g/cm<sup>3</sup> and contains an ether-soluble fraction of approx. 10 to 30%. Examples of suitable comonomers, of which 0.1 to 10 per cent by weight, relative to butene-1, can be added to the butene-1, are propene, pentene-1, hexene-1, octene-1 and dodecene, or mixtures of these  $\alpha$ -olefins with one another, but preferably propene.

The  $\text{TiCl}_3 \cdot n\text{AlCl}_3$  suitable for the mixed catalyst is preferably obtained by reduction of titanium tetrachloride with aluminium or with an organo-aluminium compound, preferably with ethylaluminium sesquichloride. The reduction with ethylaluminium sesquichloride is preferably carried out at temperatures of  $-10$  to  $0^\circ\text{C}$ . The titanium trichloride, which contains aluminium chloride, is subsequently, if desired, heat-treated at  $70$  to  $150^\circ\text{C}$ . The ethylaluminium sesquichloride is preferably employed as a 10 to 30 per cent strength solution in a hydrocarbon, for example a solution in hexane. The resulting precipitate of  $\text{TiCl}_3 \cdot n\text{AlCl}_3$  can be isolated by decanting off the liquid and washing with an inert hydrocarbon such as butane or hexane; and the resulting material may then be brought into contact with the mixture of dialkylaluminium monochloride and aluminium trialkyl. However, the suspension of  $\text{TiCl}_3 \cdot n\text{AlCl}_3$  in the liquid already contains alkylaluminium dichloride originating from the reduction, and it is possible to convert this alkylaluminium dichloride, before the polymerisation, by the addition of an appropriate amount of aluminium trialkyl, preferably aluminium triethyl, into a mixture of dialkylaluminium monochloride and aluminium trialkyl, preferably of diethylaluminium monochloride and aluminium triethyl, such as would constitute the second component of the catalyst. Such a procedure is described, for example, in Example 2. One mol of aluminium trialkyl is employed per mol of alkylaluminium dichloride to form 2 moles of dialkylaluminium monochloride. An additional quantity of 0.1 to 15 mol per cent of aluminium trialkyl must of course be added in order to obtain a mixed catalyst as required according to the invention.

The  $\text{TiCl}_3 \cdot n\text{AlCl}_3$  is suitably employed in a concentration of 0.1 to 10 mmols/l. The optimum concentration depends on the concentration of butene-1 and the impurities interfering with polymerisation. The molar ratio Al in the component (b): Ti in the component (a) is preferably 1:1 to 3:1, especially 1.5:1 to 2.5:1.

Preferably, a  $\text{TiCl}_3 \cdot n\text{AlCl}_3$  is employed which is not heat-stable, contrary to the data in German patent specification 1,209,297.

This  $\text{TiCl}_3 \cdot n\text{AlCl}_3$ , preferably  $\text{TiCl}_3 \cdot (0.3 \text{ to } 0.5) \text{AlCl}_3$ , splits off  $\text{AlCl}_3$  especially at temperatures up to  $250^\circ\text{C}$  and pressures of 1 to 760 mm Hg. A  $\text{TiCl}_3 \cdot 0.33 \text{AlCl}_3$ , which has Al, Ti and Cl in the atomic ratio of 1:3:12, splits off so much  $\text{AlCl}_3$  at temperatures up to  $250^\circ\text{C}$  and pressures of 1 to 760 mm Hg, that subsequently there is an atomic ratio Al:Ti:Cl of 1:4:15 to 1:4.7:16:5.

The polymerisation is carried out at temperatures of  $60$  to  $120^\circ\text{C}$ , preferably  $70$  to  $100^\circ\text{C}$ . If comonomers are added and if there is a fairly high proportion of aluminium trialkyls, it is preferable to polymerise at temperatures of  $60$  to  $80^\circ\text{C}$ . If the polymerisation temperatures are between  $80$  and  $120^\circ\text{C}$ , it is preferable to employ only small quantities of comonomers or none at all and only a low proportion of aluminium trialkyls.

Polymerisation temperatures higher than  $120^\circ\text{C}$ , additions of more than 10 per cent by weight of the comonomers, relative to butene-1, and proportions of aluminium alkyls higher than 15 mol per cent lead to products with excessively large ether-soluble fractions, which are unsuitable, especially for the manufacture of films, and hence are not permitted in the process of the invention. Thus a fairly large ether-soluble fraction leads to a strong adhesion of the films and to an inadmissible decrease of the yield stress values. The decrease of the yield stress values is particularly steep if larger quantities of aluminium trialkyl are employed. For example, if 20 mol per cent or more of aluminium trialkyl were added, the yield stress values would fall to approx. 20 to 30 kp/cm<sup>2</sup>.

At polymerisation temperatures below  $60^\circ\text{C}$ , products would be obtained which, when processed into films, would have poor resistance to tear propagation. In addition the advantageous solution polymerisation cannot be carried out at these temperatures.

The polymerisation can be carried out continuously or discontinuously. Hydrogen can be employed to regulate the molecular weight.

The butene-1 polymer manufactured under the conditions of the process according to the invention is particularly suitable for the manufacture of transparent films with good resistance to tear propagation, but, in addition, it is also very suitable for the manufacture of butene-1 polymer foams in general for processing by injection moulding and extrusion. In general, it has RSV values of 2.0 to 5.0 dl/g, preferably of 2.5 to 3.5 dl/g, corresponding to molecular weights of 766,000 to 2,386,000, preferably of 1,020,000 to 1,554,000. The ether-soluble fractions are between 10 and 30%, preferably between 15 and 25%, the yield stress values are between 80 and 150 kp/cm<sup>2</sup>, preferably between 90 and 120 kp/cm<sup>2</sup>, the tensile strength values are between 160 and 380 kp/cm<sup>2</sup> and the

values of elongation at break are between about 300 and 600%. These characteristics are in partial agreement with the characteristics of high pressure polyethylene. In distinction from high pressure polyethylene, the partly crystalline butene-1 polymer manufactured by the process according to the invention has substantially higher molecular weights and a higher heat distortion point. The Vicat A temperature of this butene-1 polymer is about 100 to 105°C, that of high pressure polyethylene is about 90°C. The resistance to tear propagation of a film manufactured from this butene-1 polymer is, for example, 10 to 15 kp/mm longitudinally and 20 to 28 kp/mm transversely. In contrast to this, films composed both of highly isotactic butene-1 polymer and also of a butene-1 polymer which has been brought to the same ether-soluble fraction of 10 to 30% by the working up process, have substantially poorer values of resistance to tear propagation, for example 0.4 to 2.2 kp/mm longitudinally and 24 to 28 kp/mm transversely. Films composed of such products propagate a tear in one direction at even a light load. The poorer resistance to tear propagation in these films can be both in the longitudinal direction and in the transverse direction to the direction of processing. Compared with high pressure polyethylene, the butene-1 polymer manufactured by the claimed process has, in addition to the improved heat distortion point, the additional advantage of an improved cold flow, a higher perforation resistance, lower permeability to gas and water vapour, strength values which are less temperature-dependent and an improved stress crack resistance. In addition it has an excellent extensibility. If specially stabilised, it is particularly suitable for the manufacture of degradable agricultural films. The butene-1 polymer manufactured by the process according to the invention can be

processed without problems. Films or other articles composed of this butene-1 polymer can be easily welded.

The examples set out below are intended to illustrate the process according to the invention.

#### Example 1

##### a) Preparation of a $\text{TiCl}_3 \cdot 0.5 \text{AlCl}_3$ contact catalyst.

1 mol of titanium tetrachloride (100 per cent strength) is added dropwise over the course of 6 hours with stirring to a 20 per cent strength solution of 1.4 mols of ethylaluminium sesquichloride. (molecular weight 123.7) in hexane, cooled to  $-5^\circ\text{C}$ . After a post-reaction time of 15 hours at 0 to  $+10^\circ\text{C}$ , the suspension of contact catalyst is heat-treated for 6 hours at  $150^\circ\text{C}$ . The precipitate of contact catalyst is then separated off and washed twice with hexane. 1 mol of a titanium trichloride contact catalyst of the composition  $\text{TiCl}_3 \cdot 0.54 \text{AlCl}_3$  is obtained in practically quantitative yield.

##### b) Polymerisation.

Butene-1 is polymerised with the aid of a mixed contact catalyst composed of 0.007 part by weight of this  $\text{TiCl}_3$  contact catalyst, 0.0102 part by weight of diethylaluminium monochloride and 0.00083 part by weight of aluminium triethyl in 36 parts by weight of butene-1 (98 per cent strength) at  $75^\circ\text{C}$  after adding 0.0002 part by weight of hydrogen and at a total pressure of 10 to 8 atmospheres gauge. After a polymerisation time of 6 hours the polymerisation is stopped by the addition of 0.1 part by weight of water vapour. 19 parts by weight of a polybutene-1 with the following characteristics are obtained merely by releasing the pressure on the polybutene solution:

85	RSV	4.0 dl/g	$\bar{M}_v$ : 1,830,000
	$\text{MF}_{190/5}$	1.2 g/10 mins.	calculated from the viscosity of the solution
	Density	0.9074 g/cm <sup>3</sup>	
	Ether extract	22.8%	
90	Yield stress	124 kp/cm <sup>2</sup>	
	Tensile strength	268 kp/cm <sup>2</sup>	
	Elongation at break	376%	
	Vicat A	107°C	

The film prepared from this material has the following characteristics:

95	Yield stress	longitudinally kp/cm <sup>2</sup>	218
		transversely kp/cm <sup>2</sup>	190
	Elongation at yield point	longitudinally %	16
		transversely %	16
	Tensile strength	longitudinally kp/cm <sup>2</sup>	354
100		transversely kp/cm <sup>2</sup>	276
	Elongation at break	longitudinally %	223
		transversely %	340

	Resistance to tear propagation	longitudinally kp/mm	14.2
		transversely kp/mm	25.1
	Tension impact test	longitudinally kpcm/cm <sup>2</sup>	1,025
		transversely kpcm/cm <sup>2</sup>	1,076
5	Notched-bar impact test	longitudinally kpcm/cm <sup>2</sup>	228
		transversely kpcm/cm <sup>2</sup>	234
	Perforation resistance	kp	26.7
	Welding factor		0.6

### Example 2

- 10 a) Preparation of a suspension of  
 $\text{TiCl}_3 \cdot 0.5 \text{AlCl}_3$

15 1 mol of titanium tetrachloride (100 per cent strength is added dropwise over the course of 6 hours with stirring to a 20 per cent solution of 1.4 mols of ethylaluminium sesquichloride (molecular weight 123.7) in hexane, cooled to  $-5^\circ\text{C}$ . The suspension is stirred at 0 to  $10^\circ\text{C}$  for 15 hours post-reaction time and is then heat-treated for 6 hours at  $140^\circ\text{C}$  with stirring. It contains 1 mol of  $\text{TiCl}_3 \cdot 0.5 \text{AlCl}_3$  and about 1 mol of  $\text{Al}(\text{C}_2\text{H}_5)_3\text{Cl}_2$ .

- 20 b) Polymerisation.

25 600 parts by weight of a  $\text{C}_4$  cut containing 54% of butene-1, 23% of trans-butene-2, 15% of cis-butene-2 and 8% of butane are

charged to a pressure-resistant 2 m<sup>3</sup> stirred vessel at a temperature of  $70^\circ\text{C}$ . The suspension of  $\text{TiCl}_3 \cdot 0.5 \text{AlCl}_3$  and  $\text{Al}(\text{C}_2\text{H}_5)_3\text{Cl}_2$  prepared according to the data of part (a) of this example and 1.1 mols = 0.1254 parts by weight of aluminium triethyl are added. Essentially all the  $\text{Al}(\text{C}_2\text{H}_5)_3\text{Cl}_2$  reacts with the  $\text{Al}(\text{C}_2\text{H}_5)_3$  to form  $\text{Al}(\text{C}_2\text{H}_5)_2\text{Cl}$  with some residual  $\text{Al}(\text{C}_2\text{H}_5)_3$  amounting to less than 15 mol per cent based on  $\text{Al}(\text{C}_2\text{H}_5)_3$  plus  $\text{Al}(\text{C}_2\text{H}_5)_2\text{Cl}$ . The polymerisation is carried out at  $70^\circ\text{C}$  and at a partial pressure of hydrogen of 0.1 atmosphere gauge and a total pressure of 8 to 7 atmospheres gauge. After a polymerisation time of 10 hours, 10 parts by weight of water are added with stirring. After releasing the pressure, 263 parts by weight of a polybutene-1 with the following characteristics are obtained:

	RSV	2.7 g/g	$\bar{M}_v$ : 1,122,000
	$\text{MF}_{190/2.16}$	0.9 g/10 mins.	
	$\text{MF}_{190/5}$	6.4 g/10 mins.	
	Density	0.9052 g/cm <sup>3</sup>	
50	Ether extract	19.2%	
	Yield stress	103 kp/cm <sup>2</sup>	
	Tensile strength	296 kp/cm <sup>2</sup>	
	Elongation at break	410%	
	Vicat A	$105^\circ\text{C}$	

### 55 Example 3

Butene-1 is polymerised with the aid of a mixed contact catalyst composed of 0.007 part by weight of a  $\text{TiCl}_3$  contact catalyst of the composition  $\text{TiCl}_3 \cdot 0.33 \text{AlCl}_3$  (commercially available titanium trichloride, reduced by aluminium), 0.0108 part by weight of diethylaluminium monochloride and 0.0002 part by weight of aluminium triethyl in 36 parts by weight of butene-1 (98 per cent

strength) at  $80^\circ\text{C}$  after adding 0.0005 part by weight of hydrogen and at a pressure of 12 to 8 atmospheres gauge. After a polymerisation time of 8 hours, the polymerisation is stopped by the addition of 0.1 part by weight of water vapour. 20 parts by weight of a polybutene-1 with the following characteristics are obtained merely by releasing the pressure on the polybutene-1 solution:

	RSV	3.4 dl/g	$\bar{M}_v$ : 1,499,000
75	$\text{MF}_{190/2.16}$	0.5 g/10 mins.	
	$\text{MF}_{190/5}$	3.3 g/10 mins.	
	Density	0.9046	
	Ether extract	13.9	
	Yield stress	95 kp/cm <sup>2</sup>	
80	Tensile strength	258 kp/cm <sup>2</sup>	
	Elongation at break	356%	
	Vicat A	$102^\circ\text{C}$	

### Example 4

85 Butene-1 is polymerised with the aid of a mixed contact catalyst composed of 0.012 part by weight of the  $\text{TiCl}_3$  contact catalyst pre-

pared in Example 1a), 0.024 part by weight of diethylaluminium monochloride and 0.00025 part by weight of aluminium triethyl in 40

- parts by weight of a  $C_4$  cut containing 52% of butene-1, 24% of trans-butene-2, 16% of cis-butene-2 and 8% of butane, at 90°C and a pressure of 15 to 10 atmospheres gauge. After a polymerisation time of 12 hours, the polymerisation is stopped by the addition of 0.2 part by weight of water vapour. After releasing the pressure, 18 parts by weight of a polybutene-1 with the following characteristics are obtained:

	RSV	3.0	$\bar{M}_v$ : 1,280,000
	MF <sub>190.216</sub>	0.5	
	ME <sub>190/5</sub>	3.4	
	Density	0.9050	
15	Ether extract	18.6%	
	Yield stress	98 kp/cm <sup>2</sup>	
	Tensile strength	297 kp/cm <sup>2</sup>	
	Elongation at break	415%	

#### Example 5

- Butene-1 is polymerised with the aid of a mixed contact catalyst composed of 0.006 part by weight of the titanium trichloride contact catalyst prepared in Example 1, 0.012 part by weight of diethylaluminium monochloride and 0.00025 part by weight of aluminium triethyl in 20 parts by weight of butene-1 (98 per cent strength) and 1 part by weight of propene (99 per cent strength) at 70°C and a pressure of 9 to 7 atmospheres gauge. After a polymerisation time of 5 hours, the unreacted butene-1 and the propene are released. 11 parts by weight of a butene-1-propene copolymer with the following characteristics are obtained:

35	RSV	3.2	$\bar{M}_v$ : 1,390,000
	MF <sub>190/5</sub>	3.3 g/10 mins.	
	Ether extract	21.2%	
	Yield stress	92 kp/cm <sup>2</sup>	
	Tensile strength	310 kp/cm <sup>2</sup>	
40	Elongation at break	426%	

#### Example 6

- Polymerisation is carried out in a mixture composed of 30 parts by weight of a  $C_4$  cut consisting of 48% of butene-1, 22% of trans-butene-2, 14% of cis-butene-2 and 16% of butane and 1.0 part by weight of a propene-propane mixture with a propene content of 60%, with the aid of a contact catalyst composed of 0.01 part by weight of  $TiCl_3$ , 0.3  $AlCl_3$  (commercially available  $TiCl_3$ , reduced by aluminium), 0.017 part by weight of diethylaluminium monochloride and 0.0002 part by weight of aluminium triethyl, at 70°C and a pressure of 9 to 7 atmospheres gauge. After a polymerisation time of 10 hours, 10.5 parts by weight of a butene-1-propene copolymer with the following characteristics are obtained after releasing the pressure:

60	RSV	2.9 dl/g	$\bar{M}_v$ : 1,335,000
	MF <sub>190/5</sub>	3.1 g/10 mins.	
	Ether extract	17.5%	
	Yield stress	104 kp/cm <sup>2</sup>	
	Tensile strength	247 kp/cm <sup>2</sup>	
	Elongation at break	353%	

#### WHAT WE CLAIM IS:—

1. A process for the manufacture of a partly crystalline butene-1 polymer, wherein butene-1, alone or with the addition of 0.1 to 10 per cent by weight, relative to butene-1, of a comonomer, is polymerised in a liquid phase, which is essentially all butene-1 or is a  $C_4$  fraction composed of butene-1, butene-2 and butane, at a temperature of from 60 to 120°C with a mixed catalyst consisting of (a)  $TiCl_3 \cdot nAlCl_3$  (where  $n=0.2$  to 0.6) and (b) a mixture of 85 to 99.9 mol per cent of a dialkylaluminium monochloride and 0.1 to 15 mol per cent of an aluminium trialkyl.
2. A process according to claim 1, wherein the butene-1 is copolymerised with propene as comonomer.
3. A process according to Claim 1 or 2, wherein polymerisation is carried out at a temperature of from 70 to 100°C.
4. A process according to any of claims 1 to 3, wherein a  $TiCl_3 \cdot nAlCl_3$  which has been prepared by reduction of titanium tetrachloride with aluminium or ethylaluminium

sesquichloride is employed as component (a) of the mixed catalyst.

5 5. A process according to claim 4, wherein the mixed catalyst is prepared by reduction of titanium tetrachloride with ethylaluminium sesquichloride in the form of a 10 to 30 per cent by weight solution in a hydrocarbon and the resulting suspension containing  $TiCl_3 \cdot nAlCl_3$  and ethyl aluminium dichloride is contacted with sufficient triethylaluminium to convert all the ethylaluminium dichloride to diethylaluminium chloride and provide 0.1 to 15 mol per cent of triethylaluminium based on the mixture of diethylaluminium chloride and triethylaluminium.

15 6. A process according to any of claims 1 to 5, wherein in component (a) of the catalyst n is from 0.3 to 0.5.

20 7. A process according to any of claims 1 to 6, wherein component (b) of the mixed catalyst contains 90 to 99 mol per cent of dialkylaluminium monochloride and 1 to 10 mol per cent of aluminium trialkyl.

25 8. A process according to any of claims 1 to 7, wherein diethylaluminium monochloride is used as the dialkylaluminium monochloride.

9. A process according to any of claims

1 to 8, wherein aluminium triethyl is used as the aluminium trialkyl.

10. A process according to any of claims 1 to 9, wherein the mixed catalyst has a molar ratio Al in component (b):Ti in component (a) of from 1:1 to 3:1.

11. A process according to claim 10, wherein the mixed catalyst has a molar ratio Al in component (b):Ti in component (a) of from 1.5:1 to 2.5:1.

12. A process according to any of claims 1 to 11 carried out in the presence of hydrogen as a molecular weight regulator.

13. A process for the manufacture of partly crystalline butene-1 polymer substantially as described in any of the foregoing Examples.

14. Partly crystalline butene-1 polymers when manufactured by a process according to any of claims 1 to 13.

15. A film composed of partly crystalline butene-1 polymer according to claim 14.

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